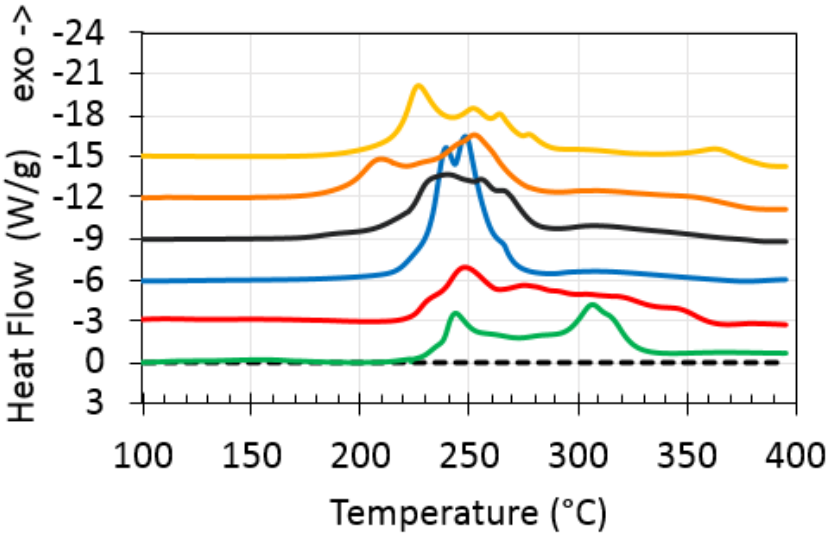
CELL ANALYSIS, MODELING, AND PROTOTYPING (CAMP) FACILITY RESEARCH ACTIVITIES



STEVE TRASK

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ARGONNE NATIONAL LABORATORY

Project ID: BAT030

2019 DOE VEHICLE TECHNOLOGIES OFFICE ANNUAL MERIT REVIEW

Washington, D.C.

June 11, 2019

OVERVIEW

Timeline

- Start: October 1, 2014
- Finish: September 30, 2020

Budget

- $850 K for FY19
  - 100% DOE-ABR

Barriers

- Need a high energy density battery for Electric Vehicle (EV) use that is safe, cost-effective, has long cycle life, and meets or exceeds DOE/USABC goals.
  - Independent validation analysis of newly developed battery materials are needed in cell formats with at least 0.2 Ah before larger scale industrial commitment

Partners

- Coordinated effort with DOE-EERE-VTO Next Generation Anodes, Next Generation Cathodes, ReCell, XCEL (INL, LBNL, NREL, ORNL, PNNL, SNL)
- Argonne Facilities: MERF, EADL, CNM & PTF
- See Collaboration list at end

RELEVANCE/OBJECTIVES

- Transition new high energy battery chemistries invented in research laboratories to industrial production through independent validation and analysis in prototype cell formats (xx3450 & xx6395 pouch cells & 18650 cells) with 20 to 3,000 mAh capacity.

- Researchers are often not able to provide the quantities of novel materials needed to make a full size EV cell to demonstrate the merits of their discoveries. The CAMP Facility is specifically designed to explore new materials with quantities as small as 50 grams for active materials, and even less for electrode/electrolyte additives.

FY19 PROGRESS MEASURES & MILESTONES

Milestone	Planned End Date	Type	Status
Complete characterization of core-shell and core-gradient materials made in MERF with focus on cycle life and thermal stability	3/30/2019	Annual	Completed
Measure electrochemical stability of LLZO ceramic produced in MERF in lithium-graphite/NMC cell environment	6/30/2019	Quarterly Progress	On-schedule
Develop methods to direct coat ceramic coatings/electrolyte membranes onto electrodes	6/30/2019	Quarterly Progress	On-schedule
Deliver needed experimental electrodes from the CAMP Facility's Electrode Library to research organizations in the U.S., including other DOE-EERE-VTO projects	9/28/2019	Quarterly Progress	On-schedule
Develop methods to fabricate pre-lithiated high-energy electrode couples on pilot-scale coater in dry room **(see BAT028)**	9/30/2019	Quarterly Progress	On-schedule

APPROACH/RESOURCES

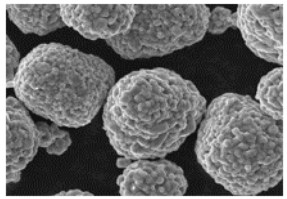
- Researchers submit materials with promising energy density
- Small hand-coated electrodes are made
- Coin cells are made and tested

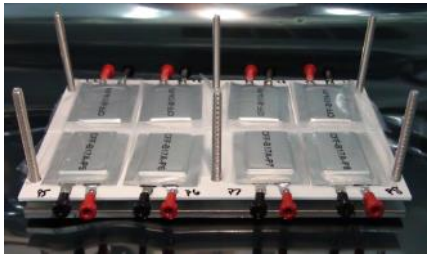
Glove box
Benchtop

- Larger material samples are obtained (MERF, partnerships, etc.)
- Longer lengths of electrode are made from scaled materials
- Pouch cells or 18650s are made and tested

Dry Room
Pilot scale

- Extensive diagnostics & electrochemical modeling on promising technologies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TECHNICAL ACCOMPLISHMENTS (NOT ALL PRESENTED AT AMR)

- Completed thermal analysis (DSC) of core-shell and core-gradient materials made in Materials Engineering Research Facility (MERF)
- Completed pouch cell life cycling of electrodes made from MERF's gradient/core-shell cathode powders **(see also BAT375)**
- Continued process development of ceramic coatings, including doped LLZO powders, on anode electrodes
- Electrochemically tested ceramic coated anodes in half- and full-cells with and without a polymer separator present
- Performed slurry and electrode development for prelithiation via the cathode electrode using Li_5FeO_4
- Continued method development for prelithiation of graphite powder & electrode via electrochemistry **(see BAT028)**
- Supported numerous DOE projects with prototype electrodes & cells
- Completed temperature study of NMC111, NMC532, NMC622, NMC811, and NCA in full cells versus graphite at -20°C, 0°C, 20°C, and 40°C for BatPaC modeling
- Completed development of a reference electrode cell setup that enables measurements of electrode potentials during fast charging **(see also BAT339 and BAT340)**
- Developed a Raman spectroscopy methodology to examine heterogeneity across the electrode cross section **(see also BAT253)**

SINGLE LAYER POUCH CELL EVALUATION OF MERF CATHODE POWDERS

Full cell formation performance of NMC core-shell (CS), core-gradient (CG), and core-multi-shell (CMS) powders from MERF yielded slightly lower discharge capacities compared to the baseline

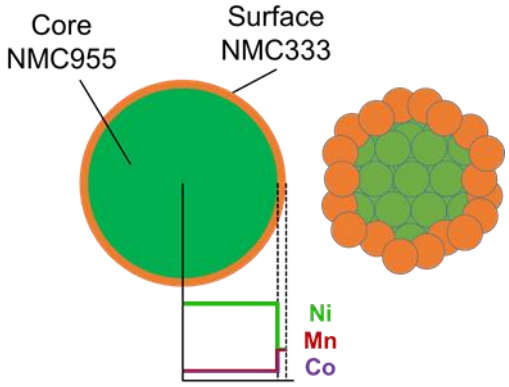
Cell Build Identifier	Average 1st Charge Capacity (mAh/g_{NMC})	Average Reversible Discharge Capacity (mAh/g_{NMC})
Targray NMC811	221	203
NMC811-CS	208	186
NMC811-CG	211	188
NMC811-CMS	221	193

1.2M LiPF6 in EC:EMC 3:7 by wt% (Gen2) electrolyte, 30 °C, 3.0 to 4.3 V, C/10

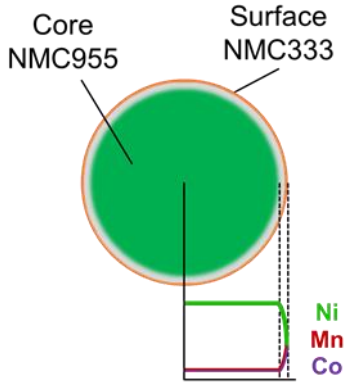
CAMP Facility:

- CS,CG, and CMS cathode powders provided by MERF, baseline by Targray
- Cathodes made to capacity match a graphite anode (A-A002B, ~2 mAh/cm²)
  - 90% active, 5% carbons, 5% PVDF
  - ~1.6 mAh/cm²
- Made 4 single-layer pouch cells for each cathode, ~20 mAh

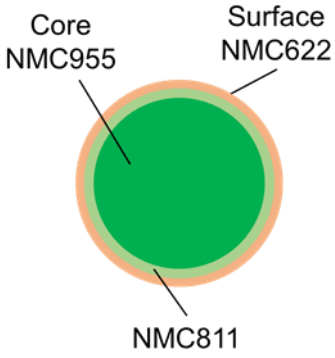
811 Core-Shell



811 Core-Gradient



811 Multi-Shell



More info at BAT375

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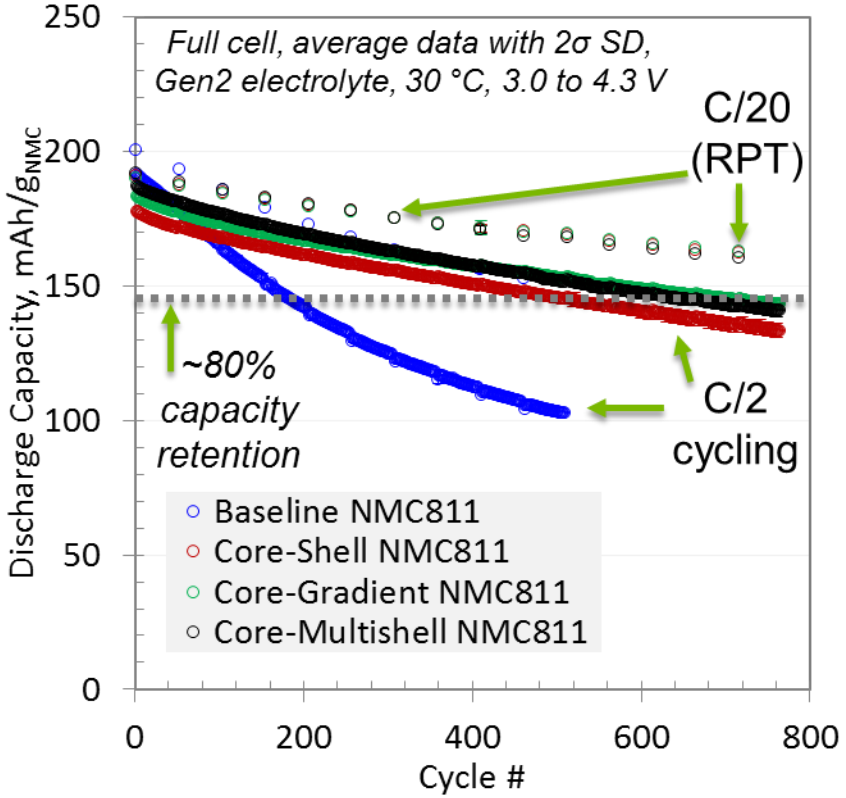
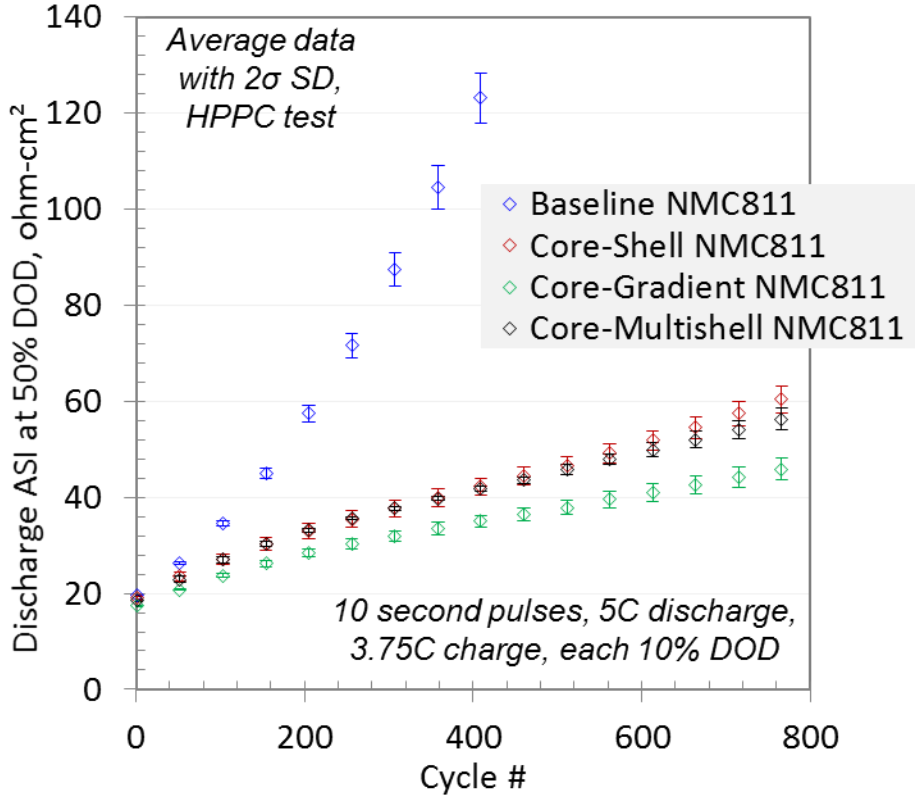
POUCH CELL CYCLE LIFE PERFORMANCE OF MERF CATHODE POWDERS





- **MERF cathode powders had slightly lower initial cycling capacities**
- **However, as the cycle life test continued, the MERF cathode powders all had significant capacity retention improvement and considerably lower impedance rise compared to the baseline NMC811**
- **The top performer for capacity retention and impedance was NMC811-CG**

More info at BAT375

THERMAL ANALYSIS OF NMC811 BASELINE AND MERF NMC811-CS, -CG, –CMS CATHODES

CAMP-made electrodes consisting of the NMC811 baseline and MERF cathode powders were tested by DSC to study the thermal stability based on decomposition temperature and heat released

Cycling - CR2032 half-cells with Gen2, 30 °C
- 3 x C/10 formation cycles, 3.0–4.5V
- Potentiostatic hold at 3.0V until C/100
- C/50 charge to *specific energy* cutoff of 750 mWh/g
  - ≈$Li_{0.24}MO_2$, 76% State-of-Charge (SOC)

Sample Preparation
- Cell disassembled in glove box
- Cathode rinsed 2 x 60s in 1mL EMC, dried 10 min under dynamic vacuum
- Cathode laminate powder scraped from current collector
- 2–3 mg cathode powder added to DSC capsule with Gen2 electrolyte (1µL:1mg powder)
- 2 cells, 2 samples per cell tested for each chemistry

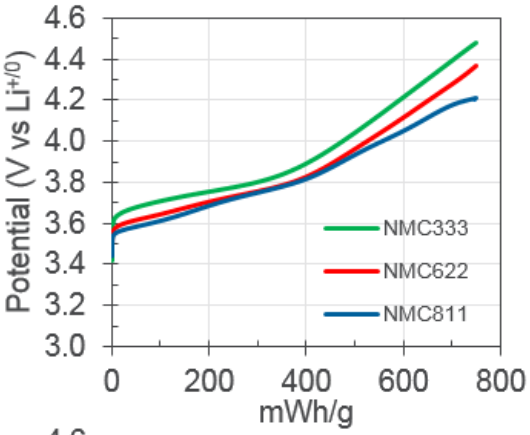
DSC Testing
- 30–400 °C, 10 °C/min.
- Baseline of empty pan subtracted from data

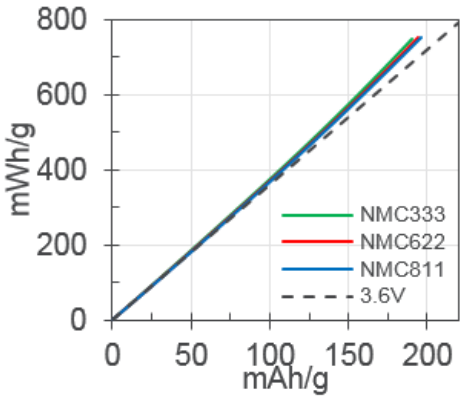
Laminate powder scraped from electrode in glovebox

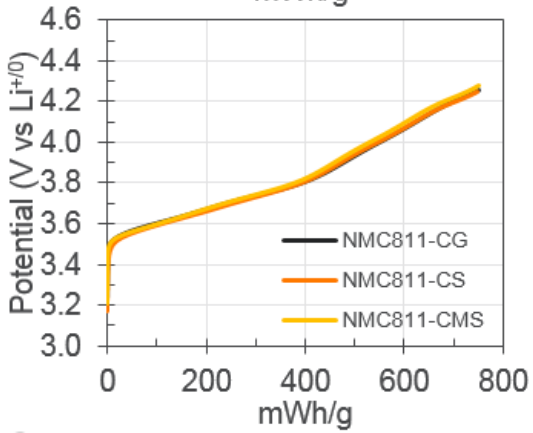
Reusable steel high pressure capsules with gold-plated caps, Perkin Elmer

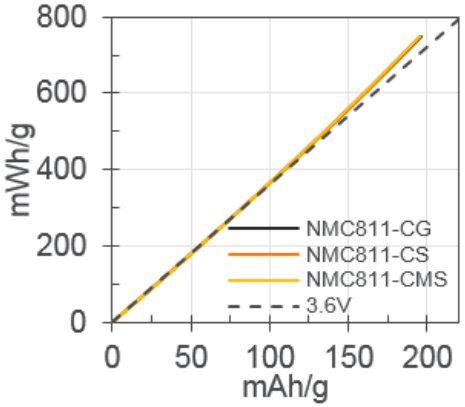
THERMAL ANALYSIS OF NMC811 BASELINE AND MERF NMC811-CS, -CG, –CMS CATHODES

Comparing cathode materials at the same charged specific energy (≈ specific capacity) rather than the same upper-cutoff voltage for a more objective analysis







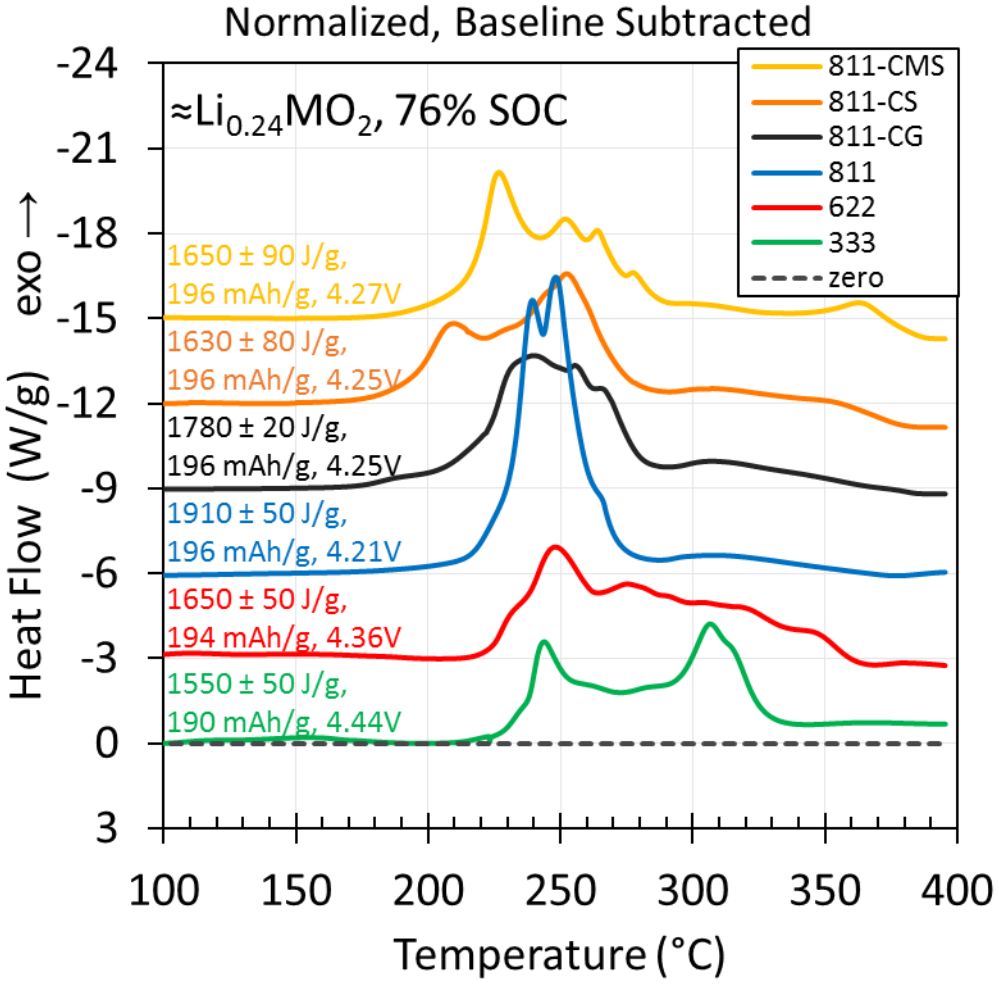


	750 mWh/g Delith. Cap (mAh/g)	750 mWh/g Delith. Voltage
NCM333	190	4.44
NCM622	194	4.36
NCM811	196	4.21
NCM811-CG	196	4.25
NCM811-CS	196	4.25
NCM811-CMS	196	4.27

- Charged (delithiated) to 750 mWh/g_{NMC}
- NMC333 and NMC622 were also included in this study to provide a broader lens for NMC thermal stability

THERMAL ANALYSIS OF NMC811 BASELINE AND MERF NMC811-CS, -CG, –CMS CATHODES

DSC results suggest the examined gradient/shell materials have mixed thermal stability results when comparing by similar charge energy



- DSC exothermic peaks associated with cathode oxygen loss and reaction with electrolyte
- MERF NMC811-CS, -CG, and -CMS materials exhibit lower decomposition onset temperature than commercial NMC333, 622, and 811 at comparable SOC

Exothermic heat released (integrated DSC peak area), half-cell charged specific capacity, and half-cell voltage before cell disassembly is listed next to the corresponding lines in plot.

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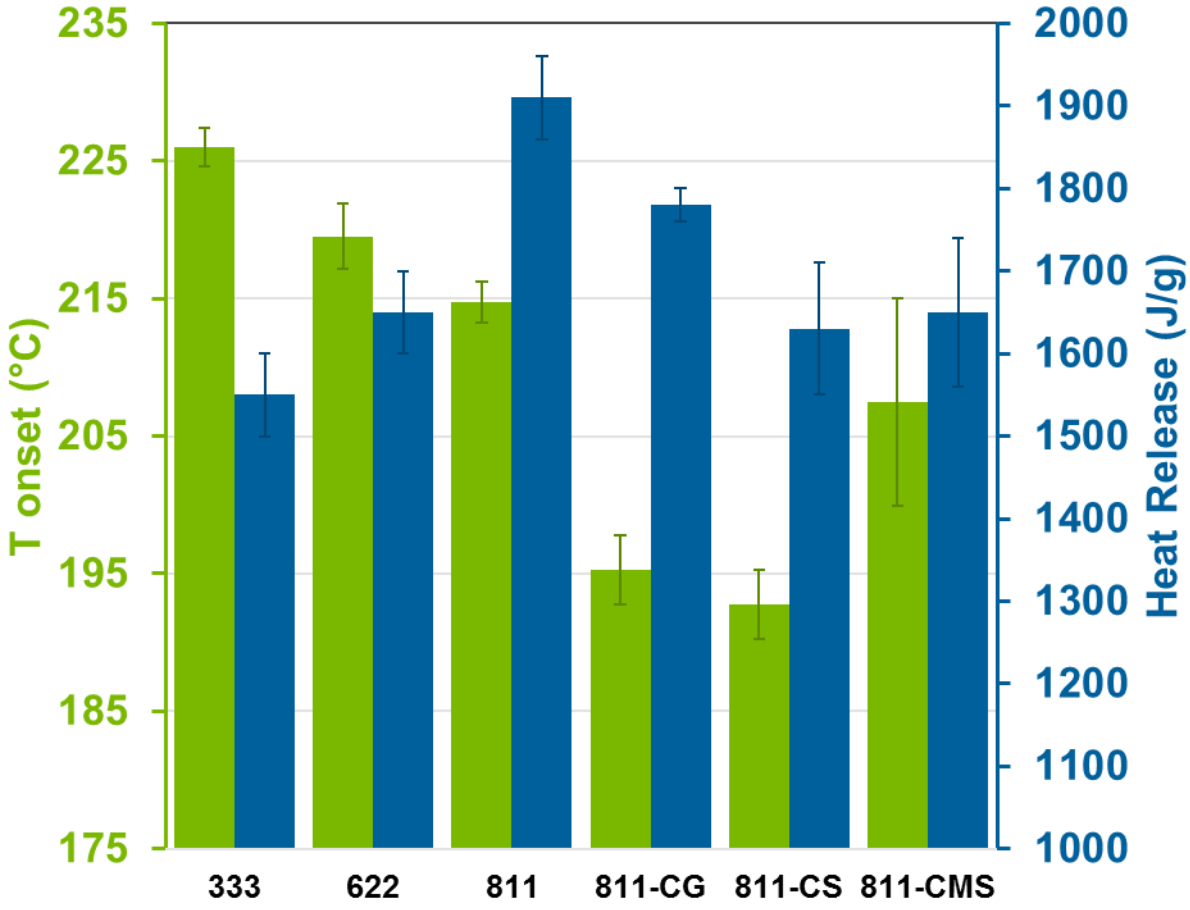
THERMAL ANALYSIS OF NMC811 BASELINE AND MERF NMC811-CS, -CG, –CMS CATHODES

- While the MERF NMC811 -CS, -CG, and -CMS materials have a lower decomposition onset temperature, they do have less total heat released over broader temperature range compared to commercial NMC811
- Thermal stability is known to decrease with increasing Ni content

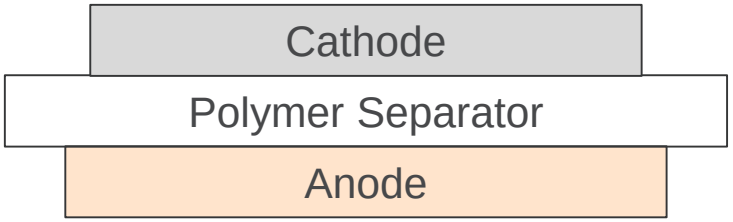


COATING SOLID FILMS ON ANODE

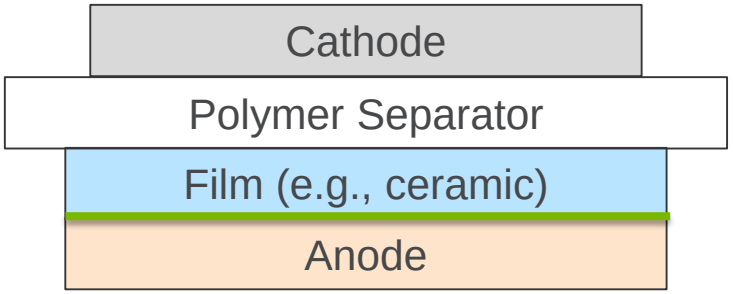
CAMP Facility continues to develop the capability to coat specialty films onto electrodes (preferably the anode). The techniques being learned will be valuable to the adaptation of processing solid electrolyte electrode systems. The effort remains on ceramic-based films, but may later include active materials or polymer films, that could serve a variety of applications:

- Replace polymer separator with ceramic separator (added safety)
- Getter undesirable decomposition products with ceramic/functional film
- Suppress formation of lithium dendrites with novel electrode architecture
- Replace liquid/polymer electrolytes with solid state electrolyte

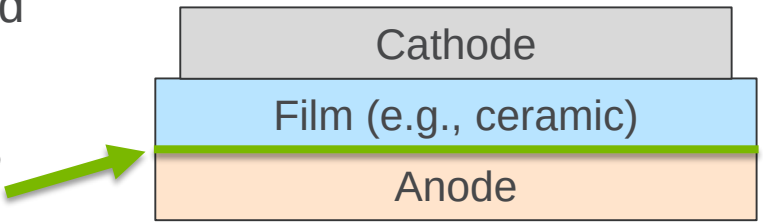
Common Cell Assembly



Scoping of Solid Film for Chemical Stability



Scoping of Solid Film for Electronic Insulation Properties

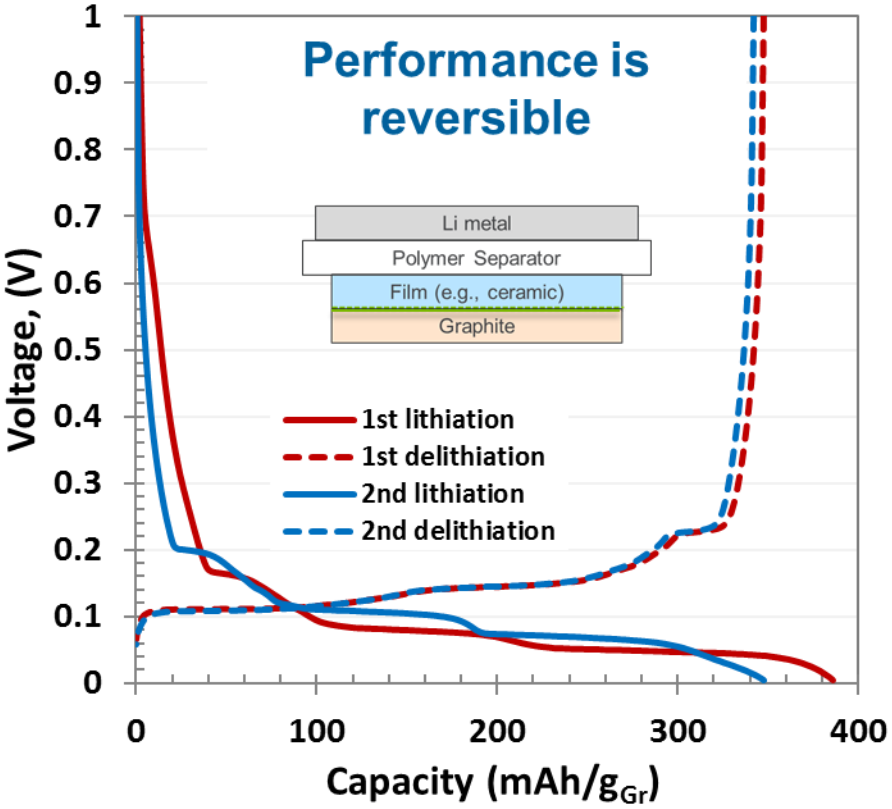


Indicates interface of film coating on to the anode

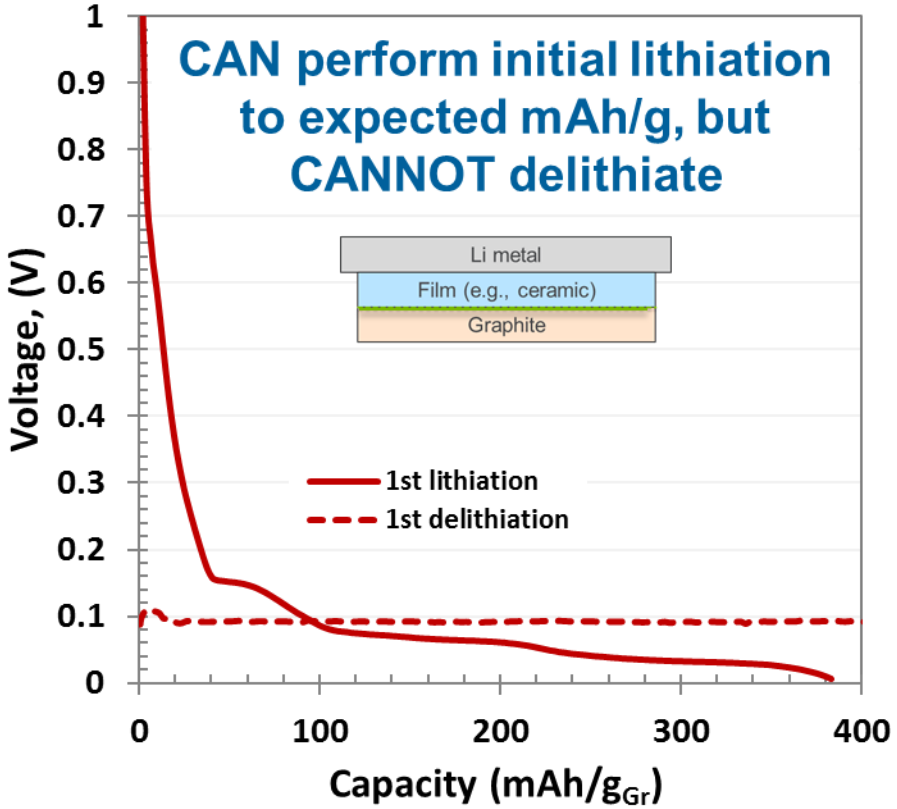
COATING CERAMIC-SEPARATORS ON ANODE

Half-cell scoping of Al_2O_3 and MgO coated graphite electrodes

WITH Polymer separator



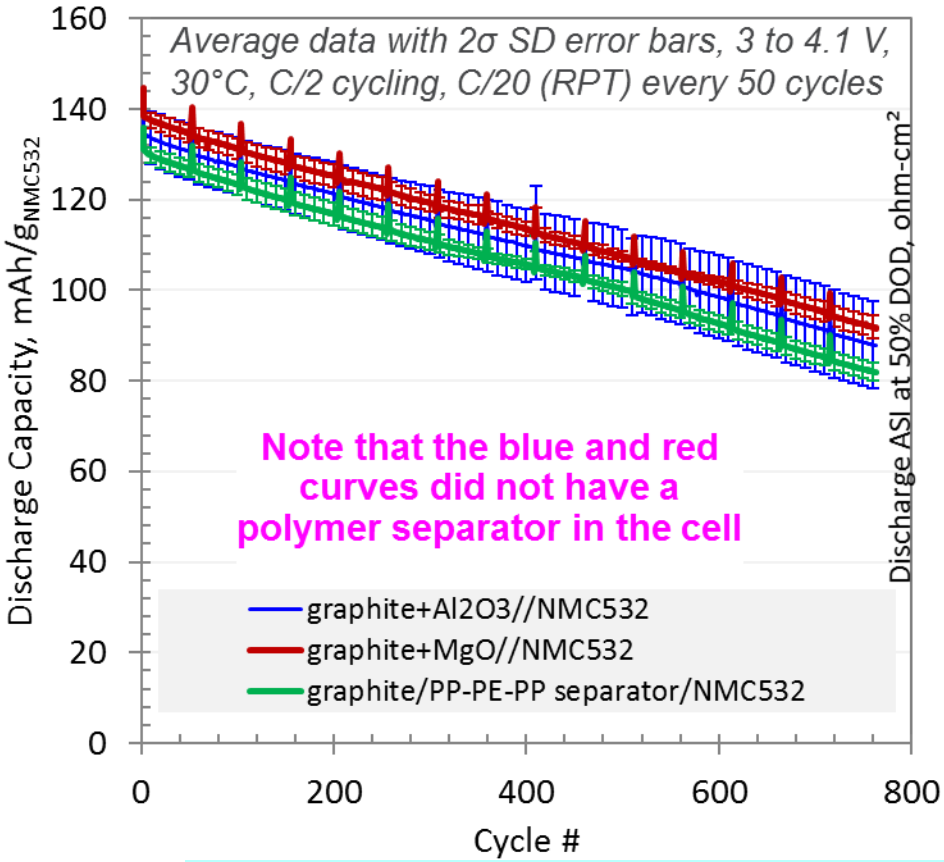
WITHOUT Polymer separator

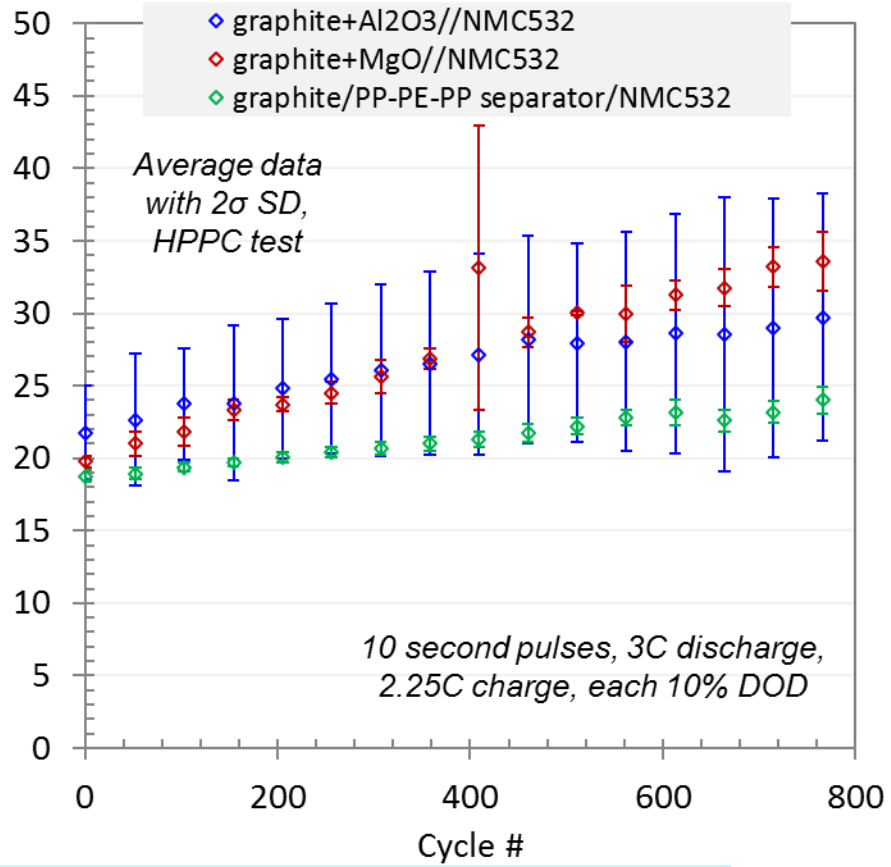


- Al_2O_3 and MgO ceramic coatings both appear chemically stable with a polymer separator between it and lithium metal
- However, the films and lithium metal show chemical instability when in direct contact, while maintaining electronic insulation properties (OCV after 1st delithiation remained constant above 0 V)

CERAMIC COATING ON GRAPHITE ELECTRODE

Completed full-cell coin-cell testing with Al_2O_3 and MgO ceramic coatings on graphite WITHOUT a polymer separator and validated the continued research of using solid ceramic separators as the sole means for electrical insulation





The coatings of Al_2O_3 and MgO onto a graphite electrode provided enough electrical insulation for the cell to not short and achieved similar performance of a typical polymer separator containing cell after ~750 cycles

CERAMICS EVALUATED

Materials below have been coated on graphite electrodes for single layer full-cell pouch cell builds

Name	Linear Formula	Molecular Weight	Description	Particle Size
Boron nitride	BN	24.82	powder, ~1 µm, 98%	~1 µm
Aluminum oxide	Al_2O_3	101.96	nanopowder, gamma phase, <50 nm particle size (TEM), alumina	<50 nm (TEM)
Aluminum silicate	$3Al_2O_3 \cdot 2SiO_2$	426.05	Aluminosilicate, Mullite	TBD
Zirconium(IV) oxide	ZrO_2	123.22	powder, 5 µm, 99% trace metals basis, Zirconia	5 µm
Magnesium oxide	MgO	40.3	≥99% trace metals basis, -325 mesh	<44 µm
Zirconium(IV) silicate	$ZrSiO_4$	183.31	−325 mesh, Zirconium(IV) silicon oxide	<44 µm
Aluminum oxide	Al_2O_3	101.96	powder, primarily α phase, ≤10 µm avg. part. size, 99.5% trace metals basis, Alumina	≤10 µm
Aluminum oxide	Al_2O_3	101.96	fused, powder, primarily α-phase, -325 mesh, Alumina	<44 µm
Aluminum nitride	AlN	40.99	powder, 10 µm, ≥98%	10 µm

Material selection based on scoping a variety of ceramics with various properties in an effort to better understand potential processing challenges that may occur with solid-electrolyte systems (i.e. LLZO)

CERAMIC MATERIALS COATED ON GRAPHITE ANODE

Compared to Celgard 2325 Separator – 25 microns – 1.53 mg/cm²

Graphite electrode was calendered before ceramic coating, then lightly re-calendered after the ceramic coating had been applied

Electrode	Cu Foil Thickness (microns)	Graphite Thickness (microns)	Ceramic Coating Thickness (microns)	Total Thickness (microns)	Graphite Loading (mg/cm²)	Ceramic Coating Loading (mg/cm²)
Baseline Graphite	10	42	0	52	6.06	0
Zirconium (IV) Oxide			35	87		9.27
Aluminum Silicate (Mullite)			40	92		5.55
Zirconium(IV) Silicate			33	85		5.67
Boron Nitride			36	88		3.64
Aluminum Oxide (10um)			36	88		7.06
Aluminum Nitride			33	85		5.60
Aluminium Oxide (comma coated)			25	77		4.29
Magnesium Oxide (comma coated)			24	76		2.51

All values are averages of samples

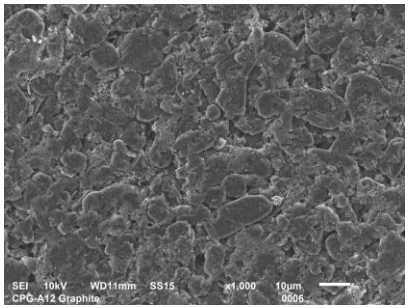
95 wt% Ceramic
5 wt% PVDF

Depending on the material chosen, these non-optimized initial ceramic coatings resulted in thicknesses of 24 to 40 µm and 2.51 to 9.27 mg/cm² loadings

CERAMIC COATING ON GRAPHITE ELECTRODE – SEM OF SURFACES

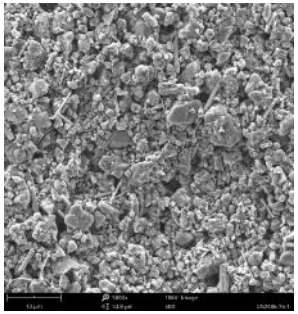
SEM images provided by Gerald T. Jeka (MERF)

10 µm scale bar



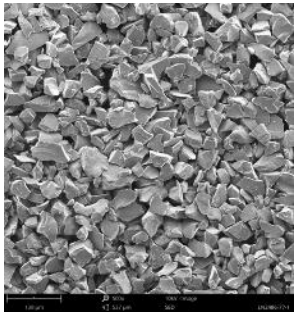
Baseline Anode

10 µm scale bar



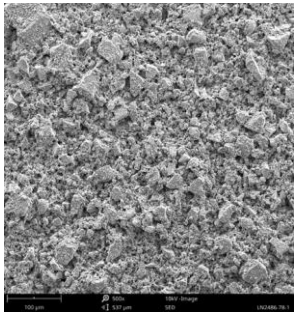
Zirconium (IV) Oxide

100 µm scale bar



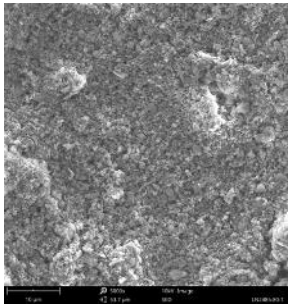
Aluminum Silicate (Mullite)

100 µm scale bar



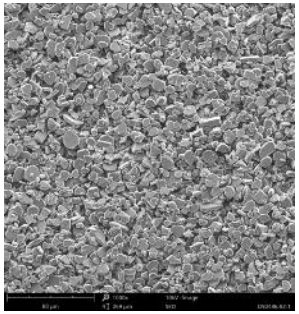
Zirconium (IV) Silicate

10 µm scale bar



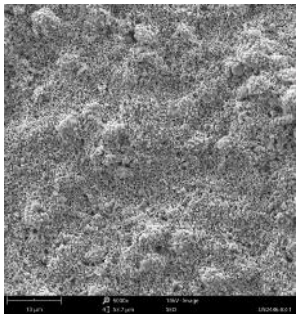
Boron Nitride

80 µm scale bar



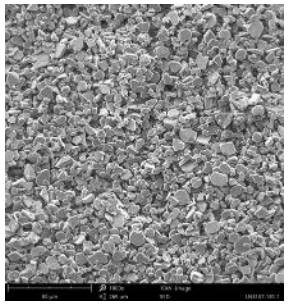
Aluminum Oxide (10 um)

10 µm scale bar



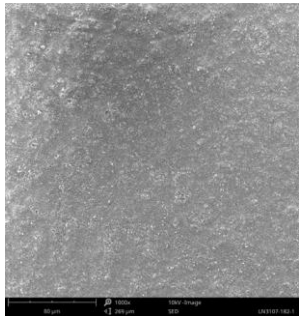
Aluminum Nitride

80 µm scale bar



Aluminum Oxide 10 um, Via comma coating

80 µm scale bar



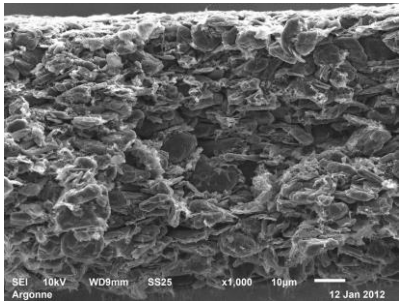
Magnesium Oxide Via comma coating

The various ceramic coatings appear to provide a uniform coverage over the surface of the calendered graphite baseline anode

CERAMIC COATING ON GRAPHITE ELECTRODE – SEM OF CROSS SECTIONS

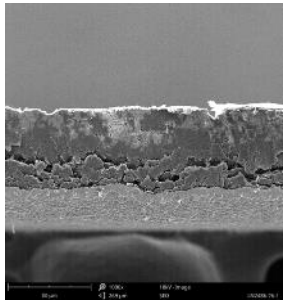
SEM images provided by Gerald T. Jeka (MERF)

10 µm scale bar

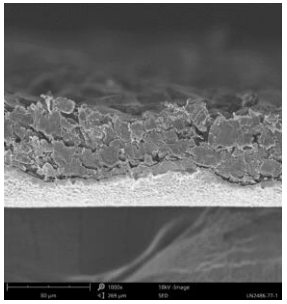


Baseline Anode

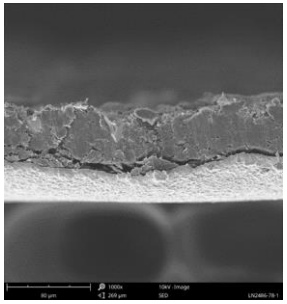
All have 80 µm scale bars



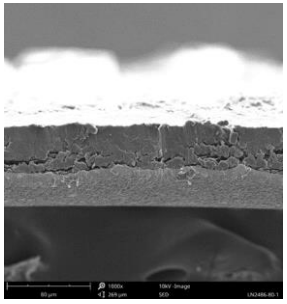
Zirconium (IV) Oxide



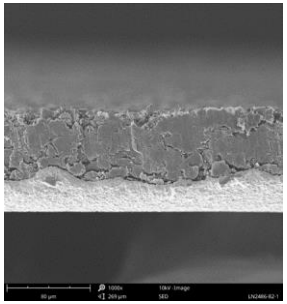
Aluminum Silicate (Mullite)



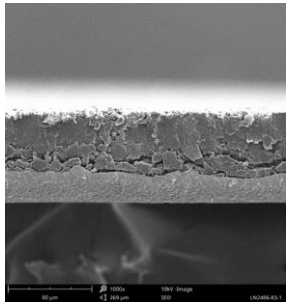
Zirconium (IV) Silicate



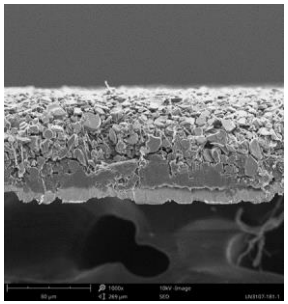
Boron Nitride



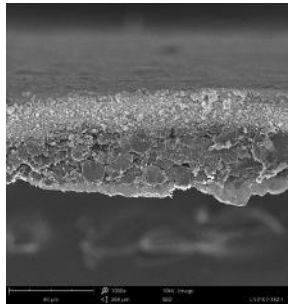
Aluminum Oxide (10 um)



Aluminum Nitride



Aluminum Oxide 10 um, Via comma coating

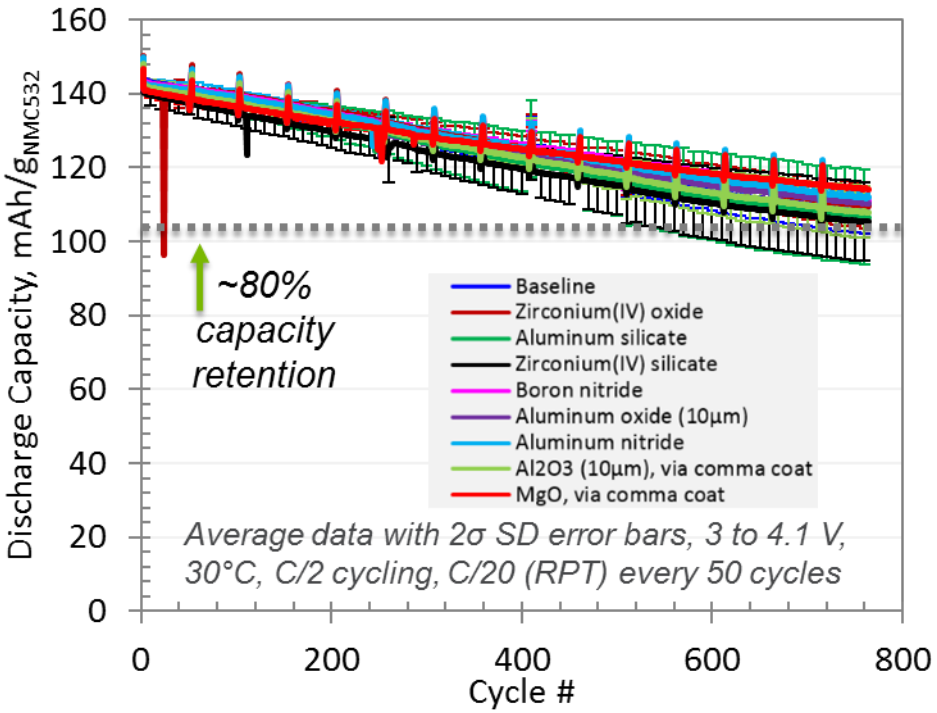


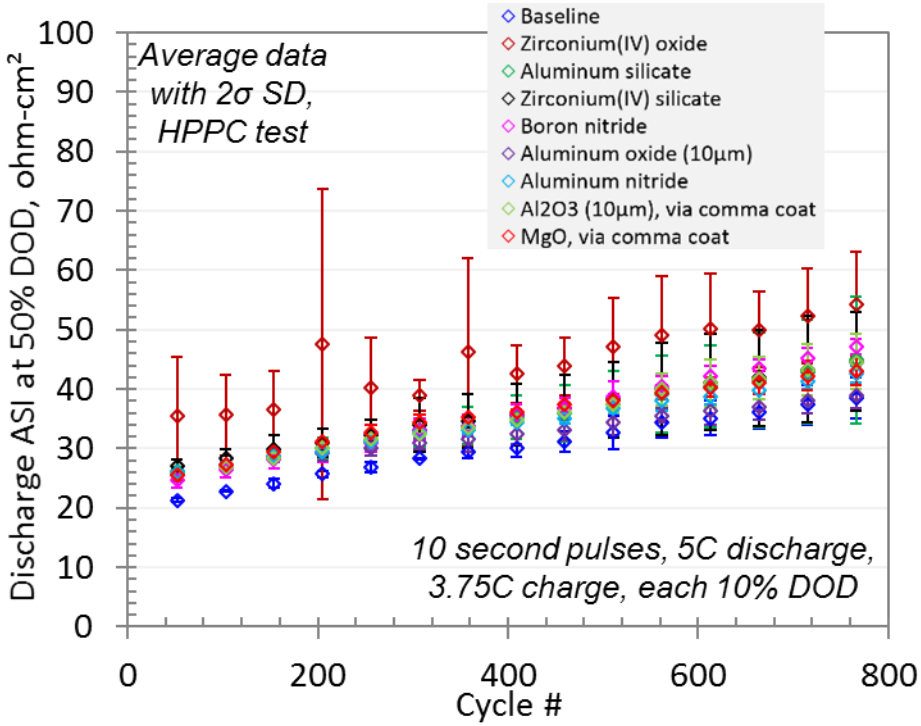
Magnesium Oxide Via comma coating

Cross section SEM images of the various ceramic coatings generally indicates a more uniform thickness is achieved using the comma coater compared to a hand coating

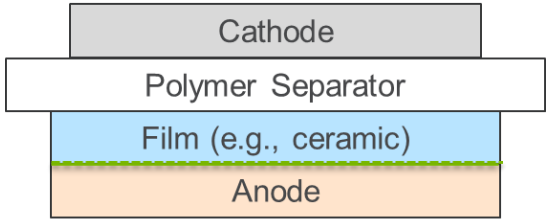
POUCH CELL ANODES WITH CERAMIC-COATING

Full cell cycle life testing indicates good ceramic compatibility with cell system





Configuration used in this pouch cell study

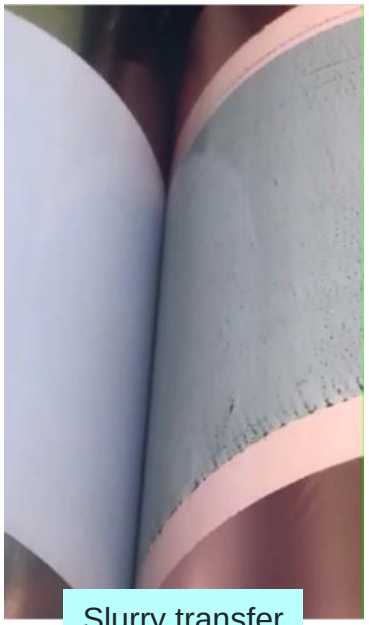


- Cell impedance is higher for the ceramic-coated electrodes, however many experience similar impedance rise rates as they are cycled
- Further investigation is needed with testing the ceramic separators without a polymer separator present in the pouch cells to better understand the pros and cons of the system

COATING HIGH QUALITY SOLID FILMS ON POROUS ANODE REQUIRES ADDITIONAL COATING EQUIPMENT

Coated with doctor blade by hand → *Reverse Comma* → *Comma* → *Future Coating Options*

Sufficient for small scale, initial evaluation



Slurry transfer challenges

Uniform, however lacks processing control

CAMP Facility pursuing new coating equipment with options including:

- Single slot die
- Dual slot die
- Gravure
- Knife-over-roll

While the comma coating method enables a uniform coating, other coating technologies provide application methods more conducive to enhancing the process

LLZO CERAMIC EXHIBITS SLURRY AND ELECTRODE PROCESSING CHALLENGES

- Purchased commercial doped LLZO materials due to the need of +100 gram powder quantities in order to understand and address possible slurry and electrode fabrication observations:
  - $Li_{6.25}La_3Zr_2Al_{0.25}O_{12}$; 250 grams
  - $Li_{6.25}La_3Zr_2Ga_{0.25}O_{12}$; 250 grams
  - $Li_{6.6}La_3Zr_{1.6}Ta_{0.4}O_{12}$; 250 grams

95 wt% Ceramic
5 wt% PVDF

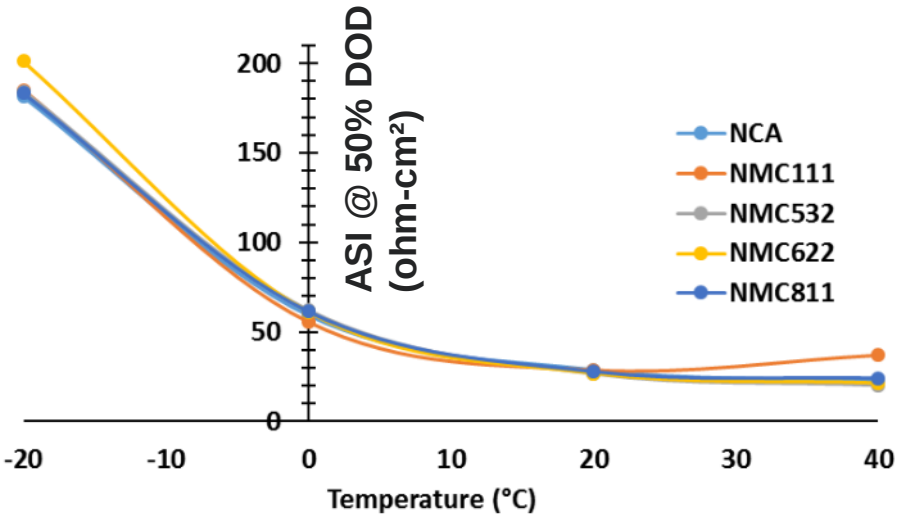
- Initial slurries indicated gelation during mixing. This result could be due to the high pH that the doped LLZO materials exhibit, which would correlate with previous observations of high pH materials when mixed with PVDF in NMP.

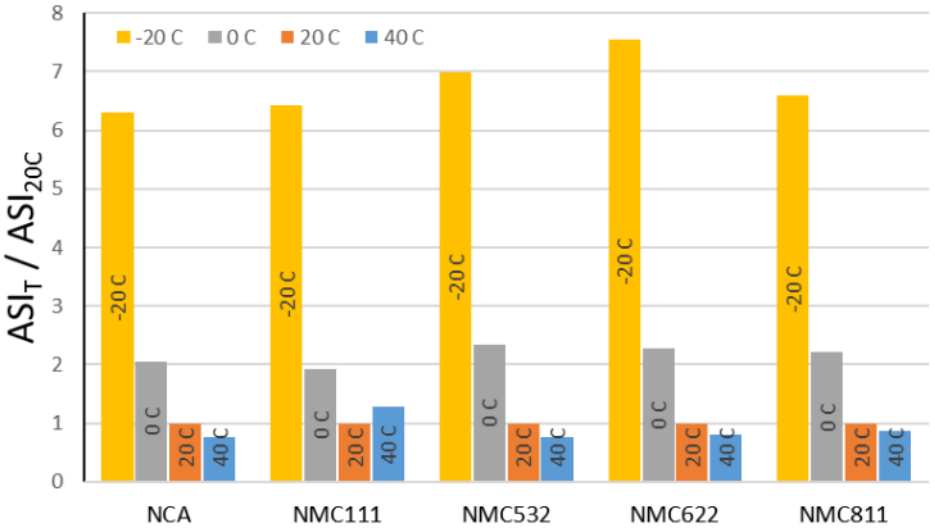
- We are investigating the possibility of pH stabilization of the LLZO powders, as well as exploring other binder and solvent systems in order to achieve improved slurry and corresponding coating properties.

- New coating system will help advance this work.

TEMPERATURE EFFECT ON CELL IMPEDANCE

ASI (area specific impedance) for NMC111, NMC532, NMC622, NMC811, and NCA in full-cell coin-cells have similar responses to each other as the temperature changes; lower temperatures result in significant changes





Individual cell builds for each temperature and cathode, 1.2M $LiPF_6$ in EC:EMC 3:7 by wt% (Gen2) electrolyte, Celgard 2320 separator

With respect to 20°C:

- 6-7X ASI rise at -20°C
- 2X ASI rise at 0°C
- Minor ASI decrease at 40°C

3 to 4.2 V, HPPC test: 10 second pulses, 3C discharge, 2.25C charge, each 10% DOD (only showing 50% DOD in plots)

Data will be fit to semi-empirical model to provide correlation for use in BatPaC

CAMP FACILITY TRIPLES ITS WORK SPACE WITH COMPLETION OF NEW DRY ROOM

The larger dry room creates the necessary space for critical new equipment needed to accomplish the CAMP Facilities R&D goals

Argonne welcomed DOE VTO Director Michael Berube and Deputy Director David Howell in September 2018 to the ribbon-cutting ceremony of the CAMP Facility's new dry room.

The new lab space was generously funded by Argonne and the Chemical Sciences and Engineering (CSE) Division at Argonne.

From Left: Argonne Laboratory Director Paul Kearns, Argonne CSE Division Director Cynthia Jenks, DOE-VTO Director Michael Berube, VTO Deputy Director Dave Howell, and CAMP Facility Team Leader and Group Leader Andrew Jansen

Photo by Wes Agresta (CPA-CR, Argonne)

REVIEWER COMMENTS FROM 2018 ANNUAL MERIT REVIEW

- No reviewer comments from 2018 Annual Merit Review

CAMP FACILITY'S ELECTRODE LIBRARY SERVES THE BATTERY COMMUNITY

- The Electrode Library serves as a supply of standard electrode samples that are designed to be interchangeable with one another (capacity matched)
- Electrodes can be made with as little as 50g of experimental material, and can be made to match an existing counter electrode

Currently Available:
- 17 anodes
- 22 cathodes

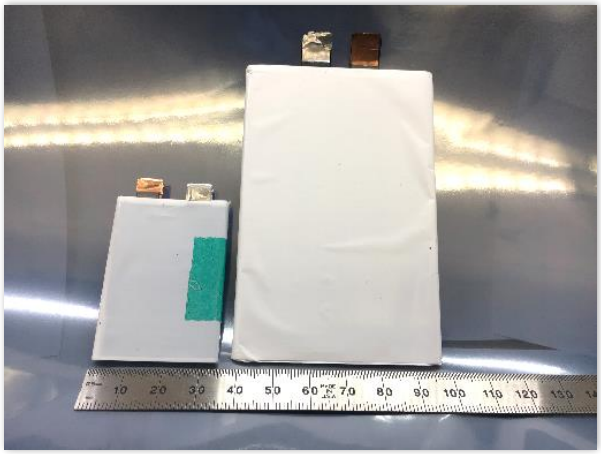
- Neg:Pos balanced
- Anodes at 2 mAh/cm^2

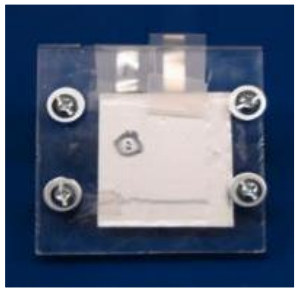
- 220 mm x 110 mm of coating per sheet

Electrodes Delivered	FY16		FY17		FY18		FY19 (as of April 2019)	
Argonne	174	8 %	142	9 %	140	14.3 %	62	12.4 %
Other National Labs	726	36 %	172	11 %	172	17.5 %	47	9.4 %
Universities	117	6 %	151	10 %	175	17.8 %	152	30.5 %
Industry	1004	50 %	1083	70 %	495	50.4 %	237	47.6 %
Total:	**2021**		**1548**		**982**		**498**	

CAMP FACILITY DELIVERS POUCH CELLS FOR THE BATTERY COMMUNITY

- Pouch cells provide critical information on the scalability of newly developed battery materials
- CAMP pouch cell format capabilities of xx3450 and xx6395, as well as customized design to meet researchers' needs in unique experimentation
- xx3450 cells range from single-layer (~20 mAh) to multi-layer (~600 mAh)
- xx6395 cells range from single-layer (~70 mAh) to multi-layer (~3000 mAh)





Electrodes Delivered	FY16		FY17		FY18		FY19 (as of April 2019)	
Pouch Cells Delivered	29		55		162		123	

Argonne NATIONAL LABORATORY

CAMP FACILITY COLLABORATIONS

Majority of these collaborations over the past several years are centered on the CAMP Facility providing electrodes and cells

Argonne NATIONAL LABORATORY

REMAINING CHALLENGES/FUTURE WORK

- Direct coating roll-to-roll options needed to effectively coat ceramic materials onto (solid-separator) and in electrodes (solid-electrolyte)
  - Application of films via single slot die, dual slot die, gravure, knife-over-roll, etc. are needed to be explored
- Determine strategies to overcome non-ideal rheological slurry/coating behavior when they are encountered (gelation, phase separation, agglomerates, aggregates, etc.)
  - Use gained processing knowledge from specialty coatings to other materials such as active materials or polymer films
- Coordinate with Post-Test Facility on tested ceramic-coated anodes from pouch cells to inspect the interface between anode and ceramic layer determine possible failure mechanisms in cells and determine the value of testing pouch cells with relying on the ceramics as the sole electronic insulator (without polymer separator present)
- Complete Li_5FeO_4 (LFO) effort in testing concept with optimum concentration in pouch cells
  - Determine influence of residual iron species on energy density and cycle life
- Continue work with MERF on demonstrating performance of new cathode materials, esp., high energy, low cobalt powders
- Continue in the develop of methods to fabricate pre-lithiated high-energy electrode couples on pilot-scale coater in dry room
- Any proposed future work is subject to change based on funding levels

SUMMARY

- Completed study on core-shell, core-gradient, and core-multi-shell cathode powders from MERF by performing DSC on the electrodes and examining the cycle life performance in pouch cells
- Continued testing of ceramic powders on graphite anodes and assembled in coin-cells and single-layer pouch cells
  - Identified slurry and electrode processing challenges when incorporating LLZO powders with PVDF in NMP
  - Recognized the need of more coating methods (i.e. single slot die, dual slot die, gravure, knife-over-roll) in order to apply films/coatings onto electrodes
- Determined a 6-7X ASI rise from 20°C to -20°C in cell impedance and a 2X rise from 20°C to 0°C for NMC111, NMC532, NMC622, NMC811, and NCA in full cells
- Supplied numerous experimental electrodes and cells to DOE programs

CONTRIBUTORS AND ACKNOWLEDGMENTS

Argonne

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- David Wood III (ORNL)
- Jason Zhang (PNNL)

Research Facilities

- Materials Engineering Research Facility (MERF)
- Post-Test Facility (PTF)
- Electrochemical Analysis and Diagnostic Laboratory (EADL)
- Center for Nanoscale Materials (CNM)
- Advanced Photon Source (APS)

Industry

- Paraclete Energy
- Celgard
- Superior Graphite
- Targray

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PUBLICATIONS/PATENTS

1. J.A. Gilbert, V.A. Maroni, Y. Cui, D.J. Gosztola, D.J. Miller, D.P. Abraham, “Composition and Impedance Heterogeneity in Oxide Electrode Cross-sections detected by Raman Spectroscopy”, Adv. Mater. Interfaces (2018), 1701447.
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9. Capacity Fading Mechanism of The Commercial 18650 LiFePO4 Based Lithium Ion Batteries: An In-Situ Time-Resolved High-Energy Synchrotron XRD Study, Qi Liu, Yadong Liu, Fan Yang, Hao He, Xianghui Xiao, Yang Ren, Wenquan Lu, Eric Stach, and Jian Xie, ACS Appl. Mater. Interfaces, (2018), 10 (5), pp 4622–4629.

CRITICAL ASSUMPTIONS AND ISSUES

- Material developers will be willing to openly share their material and data with researchers funded within DOE programs. This has been an ongoing concern for several years. A few companies have been receptive to having their advanced materials available to national labs and universities, while others see no incentive to do so for fear of losing Intellectual Property rights. The CAMP Facility is often limited to commercially available materials from vendors.

- Discoveries made by material inventors at the coin cell level will be realized upon scale up to CAMP prototype cells. This is not always the case – on occasions positive results seen for thin electrode coin cells do not translate well into larger cell formats. This underlines the importance of the CAMP activities in assessing the true impact of discoveries.